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Final Report on
DEVELOPMENT OF INTERCALATED WIRE & CABLE

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TABLE OF CONTENTS

Abstract

I General Introduction

II Experimental Work

A - Arsenic Pentafluoride in Highly Oriented Pyrolytic Graphite

1. Materials
2. Procedure
3. Results

B - Antimony Pentafluoride - Graphite Cored Wires

1. Procedures
 - a) Ampoule Geometry
 - b) Filling Techniques
2. Results

III Conclusion

IV References

V Addendum

Review of Japanese work confirming ultra high electrical conductivity in arsenic pentafluoride intercalated graphite.

ABSTRACT

The purpose of this program was to confirm previously reported high electrical conductivity reported in intercalated graphite in Highly Oriented Pyrolytic Graphite (HOPG) (1) and in copper sheathed composite wires with intercalated graphite cores (2). The method employed in both of these experimental sets was to first, give careful attention to experimental details and second, to reproduce the essential details employed before in the successful demonstrations. While some very low electrical resistivities were measured, none lower than that of copper were observed.

I. GENERAL INTRODUCTION

Current engineering technology depends to a large extent on the conduction of electricity, and in the military context the weight of the conductor is important whether it is borne by personnel or high performance helicopters. Thus, there is a premium on light weight, high conductivity materials that can be used in practical applications. Silver, the best metallic conductor known, and copper, the engineering standard for electrical conduction, both suffer from relatively high densities (10.49 gm/cm^3 and 8.95 gm/cm^3 respectively). On the other hand, some lighter metals such as aluminum (2.7 gm/cm^3) or even sodium, while sometimes used or seriously considered (1) as conductors have been found unsuitable in one way or another. So, when it appeared that intercalation compounds of graphite offered the potential of light weight practical conductors, an important goal seemed within reach.

There is a good physical basis for the expectation that intercalated graphite should exhibit extraordinarily high electrical conductivity which can be explained with the following equation:

$$\sigma = ne\mu$$

where σ = electrical conductivity in $\text{ohm}^{-1} \text{ cm}^{-1}$

n = number of charge carriers per cm^3

e = electronic charge

μ = carrier mobility in $\text{cm}^2 / \text{volt} - \text{sec}$

In words, the electrical conductivity depends on the density of charge carriers times the charge on the electron times the mobility of the carriers or the ease with which they move through the metal crystal lattice. Table I below lists their values for certain materials.

Table I - Electronic Transport Characteristics of Selected Materials

<u>Material</u>	<u>Conductivity</u> <u>ohm⁻¹cm⁻¹</u>	<u>Charge Density</u> <u>no. / cm³</u>	<u>Mobility</u> <u>cm² / volt - sec</u>
Copper	5.9 X 10 ⁵	10 ²	35
Graphite a direction	0.3 X 10 ⁵	10 ¹⁹	12,000
Intercalated Graphite	3.0 X 10 ⁵	10 ²¹	2,000

From this table we see that copper owes its high electrical conductivity to the fact that its carrier (electron) density is very high - one per atom in the crystal lattice. Notice also that its carrier mobility is relatively low as a result of frequent collisions of the carriers with the random thermal motions of atoms in the metal lattice. Graphite on the other hand, has an extraordinarily high carrier mobility because collisions are less frequent in its rigidly bonded layer plane lattice. When graphite is intercalated, the intercalate molecules separate the rigid graphite layers and donate charge to the host lattice, increasing the charge density by two orders of magnitude, but decreasing the mobility by a relatively small amount. Were it possible to increase the charge density in graphite by the stated amount and reduce the mobility by a lesser amount, a truly superior conductor would result.

The intercalated graphite research group at the University of Pennsylvania have on several occasions observed and reported electrical conductivity in graphite intercalates with Lewis Acid fluorides which were equal to or greater than that of copper (2, 3, 4, 5). The first of these was the experiment by Vogel (2) in which essentially a copper ampoule approximately 0.8 cubic diameter was filled with SbF₅ - intercalated graphite sealed and swaged into a wire less than 2 mm in diameter. The composite resistivity of this wire was 1.68 X 10⁻⁶ Ω cm - slightly lower than the resistivity of copper, which is 1.71 X 10⁻⁶ Ω cm, and when the conductive effect of the sheath of the composite and the density of the core were corrected for the resistivity of the intercalated graphite core was 1.0 X 10⁻⁶ cm. In an independently executed repeat of that experiment, Gan (3, 6) obtained substantially the same result which is given in greatest detail in an earlier report (7) of this series. The most convincing aspect of Gan's work was the fact that the conductivity of the composite wire consisting of a copper sheath and intercalated graphite core was greater than the conductance of the solid copper control sample of the same diameter.

The engineering nature of the composite wire experiments just described lacked rigor in the mind of certain members of the graphite intercalation research group and so they set out to study the electronic transport of these materials using HOPG (highly oriented pyrolytic graphite) (8) and AsF_5 as the intercalate. This series of experiments resulted in a number of high conductivity samples (Figure 1 in Ref. 5) equivalent to that of silver as well as the indication (Figure 1 Ref. 5) that at a given stage, a superior conductivity can be achieved.

The experiments reported here were undertaken for two purposes: to replicate as carefully as possible, 1) the experiments of Falardeau, Foley & Zeller on AsF_5 intercalated HOPG and 2) the experiments of Gan that produced a high conductivity composite wire.

II. EXPERIMENTAL WORK

A - Arsenic Pentafluoride in Highly Oriented Pyrolytic Graphite

1 - Materials

The graphite used in this part was highly oriented pyrolytic graphite (HOPG) which was obtained from Union Carbide and used without further purification. HOPG, also known as aligned graphite, is created by simultaneously subjecting epitaxial carbon to high temperature and uniaxial stress. The carbon deposit is obtained from pyrolytic decomposition of hydrocarbon gases, principally CH_3 . The plates of graphite obtained from this process exhibit excellent alignment of the crystallographic c-axis. It has been established that with proper annealing the electrical and mechanical properties of HOPG approach those of natural single crystals (8).

Although single crystal graphite is rare, it has been extensively studied and the band structure is well known. Since the properties of HOPG are so close to those of single crystal graphite, the effects of intercalation in compounds made from HOPG can be realistically analyzed in terms of single crystal material. This is crucial to a broad investigation of the properties of intercalation compounds because natural single crystal graphite is not readily available in the necessary sizes or quantities.

Pieces of HOPG were obtained from Dr. Arthur Moore of Union Carbide (9). These were from Union Carbide's regular production of aligned graphite for x-ray monochromators, in which temperatures of 3000°C and pressures of 500 kg/cm^2 were used. The spread in c-axis orientation is typically 1° , and crystallite sizes are typically a few microns (10).

The arsenic pentafluoride was obtained from Ozark Mahoning⁷ and was checked for purity by vapor phase molecular weight determinations. In most instances it was found acceptable as received ($\pm 1\%$ of theoretical value 169.19); when unacceptable, it was purified by trap-to-trap distillation and rechecked.

2 - Procedures

The graphite samples to be intercalated were cut to 5 X 8 mm² c-face rectangles using a 0.025 cm diamond string saw. Additional samples from 5 X 5 mm² to 6 X 20 mm² were also intercalated; many of these were cut to size using air abrasion instead of the string saw. The initial thickness of most samples was in the range 0.025 to 0.064 cm; these c-axis thickness measurements were made using a microscope with a calibrated reticule (sensitivity = 5×10^{-4} cm). The initial weight of the samples fell in the range 15 to 50 mg (± 0.05 mg).

All volatile materials were manipulated utilizing standard high vacuum techniques in a glass system equipped with glass-Teflon valves (11). When necessary, intercalated materials were handled under a dry nitrogen atmosphere since the lowest stage compounds exhibited extreme sensitivity to moisture. Handling was kept to a minimum.

At the start of the program the vacuum rack used for the intercalations which had been used in a previous program (12) was rebuilt completely to assure tightness. The criterion for successful operation was taken as pressure of 0.1 μ using a liquid nitrogen trapped oil diffusion pump.

Each side arm valve was tested and replaced where necessary. Ground joints found to be leaky were sealed semi-permanently with Apiezon wax.

The AsF₅ graphite intercalation compounds were prepared in an inverted h shaped Pyrex reactor equipped with a glass Teflon valve on the sidearm (Figure 1). The main reactor tube was made of 1/2" O.D. glass, the top of which was fitted with a Swagelok SS plug fitted with Teflon ferrules. This arrangement allowed easy insertion of the solid graphite reactant and removal of the intercalated product from an otherwise closed system. The typical total volume of these reaction vessels was 10 ml. Reactions containing up to 3.6 atmospheres of AsF₅ were successfully executed without leakage through the Swagelok connection.

Each side arm valve was tested and replaced where necessary. Ground joints found to be leaky were sealed semi-permanently with Apiezon wax.

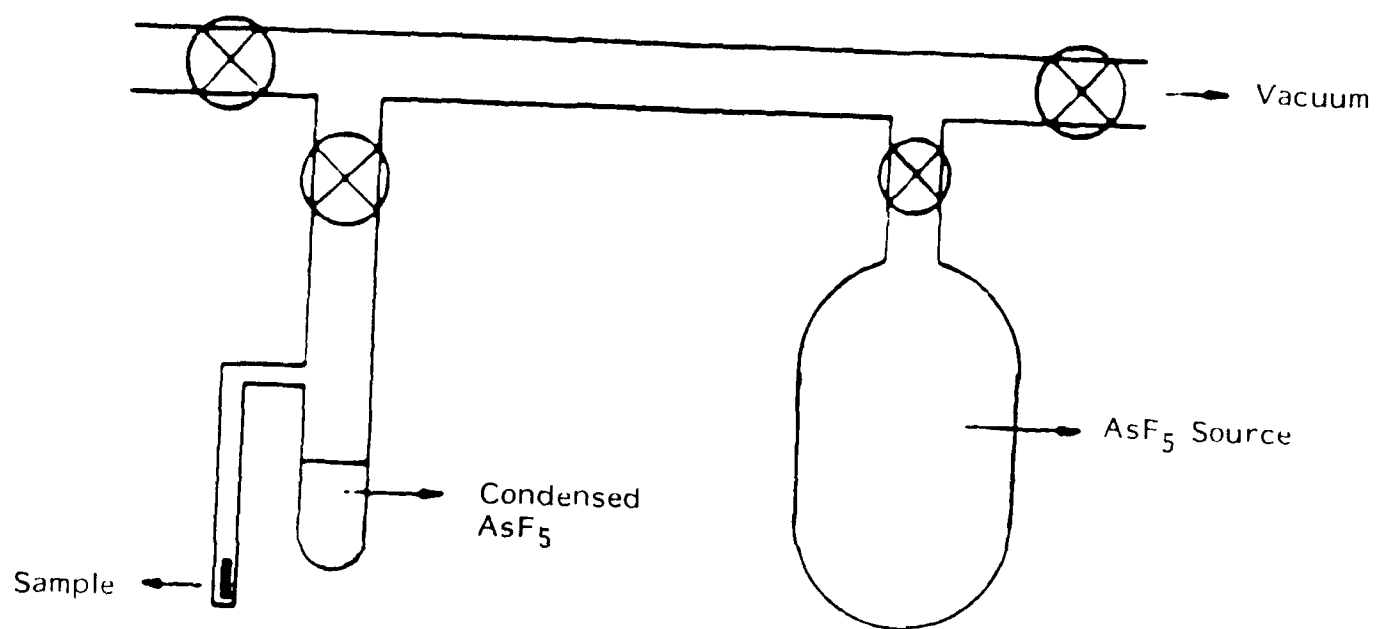


Figure 1. Side arm reactor for AsF_5 intercalation.

To produce a compound, the reactor was first carefully cleaned and baked dry in an oven. After it was removed and allowed to cool to room temperature, a freshly cleaved 5.5 X 8 X 0.5 mm piece of HOPG was placed diagonally in the sample arm. The Swagelok cap was installed, then the entire reactor was heated in a gas flame while under dynamic vacuum. After the reactor cooled to room temperature, a measured amount of purified AsF_5 was condensed into the small arm at -196°C . Typically, 0.5 to 1.5 mmole ± 0.03 mmol were used. Since only the bottom of the vessel was cooled, the graphite did not vary appreciably from room temperature. The reaction was started by allowing the AsF_5 to warm to 23°C , at which temperature it is all in the vapor state. The initial warming period mentioned above, during which the AsF_5 warms from -196°C to 23°C , was typically less than 2 minutes. No observable reaction (thickness) occurred during this time. For a typical 35 mg sample and initial pressure of 1 atm, the final pressure was 0.5 atm over a second stage compound. Where possible, measurements of the sample's thickness were made at intervals of approximately ten minutes in order to track the extent of intercalation. These thickness measurements were done using a microscope with a calibrated reticule. For samples that were transferred, when the desired stage was achieved the compound was removed, weighed, x-rayed, and transferred to the measurement ampoule. Reactions were stopped by opening the vessels in a nitrogen atmosphere and removing the intercalated graphite from the system. This method was utilized as opposed to ambient temperature vacuum removal of the excess AsF_5 because the intercalated samples of the lower stage tended to severely exfoliate (essentially explode physically) under vacuum.

Initially, for conductivity measurements the sample of graphite to be intercalated was supported in the reactor in a loose Pt wire spiral which performed two functions. First, it held the graphite in a stable position such that consistent thickness measurements could be made in situ. Second, it suspended the graphite above the bottom of the reactor. This second function enabled the intercalation process to proceed entirely in the vapor phase without contact with AsF_5 liquid during the initial warming process.

Gravimetric coupled with the c-axis thickness measurements were used to characterize the resulting compounds. Gravimetric analyses were made by weighing the intercalated samples while enclosed in preweighed Teflon containers.

In the first experiment the sample (82-8) in one side arm was baked at 150°C , while the twin of the first (82-1) was not. The baked sample gave a lower electrical resistivity and so baking was adopted as a standard procedure.

3 - Results and Discussion

The following table give the results of the first six experiments on the vapor intercalation of HOPG with AsF₅. All resistivity values were corrected to 22°C.

Table II
Electrical Resistivity of Graphite Crystals After Intercalation with AsF₅ at Various Pressures

Sample No.	Graphite Resistivity $\mu\Omega\text{ cm}$	AsF ₅ Pressure mm	Stage No.	Intercalated Resistivity $\mu\Omega\text{ cm}$
82-1	41.9	20	>10	7.1
82-2	39.5	500	2	3.0
82-3	38.8	500	2	2.9
82-4	38.1	20	>10	6.5
82-5	41.1	500	2	4.4
82-6	45.5	500	2	2.6

These results are all consistent with the lower vapor pressure yielding higher stage compounds and the lower stage compounds (stage 2) showing the lower resistivities. However, none of the stage 2 resistivities in Table II are equivalent to the very low values previously reported (4, 5) for stage 2 compounds with AsF₅.

There is some question about the interaction of AsF₃ and AsF₅ in the process of intercalation of graphite since AsF₃ is one of the products resulting from the treatment of graphite with AsF₅. AsF₃ is liquid in the range of temperatures from -59°C to -56.3°C, whereas AsF₅ is liquid between -79.8°C and -53°C. Thus, in an experiment having a cold trap held in the range -5°C to -25°C, AsF₃ pressures would be held at a low value (as, incidentally, would be the pressure of HF, if present).

Four samples were intercalated, introducing AsF₅ to the reactor at 500 mm of pressure and trapping at various sub zero temperatures with the following results:

Table III
Electrical Resistivities of Graphite Crystals after Intercalation with
AsF₅ at 500 mm Pressure with Various Trapping Temperatures

Sample No.	Graphite Resistivity $\mu\Omega\text{ cm}$	Tray Temp $^{\circ}\text{C}$	Stage No.	Intercalated Resistivity $\mu\Omega\text{ cm}$
82-7	46.4	-11	2	3.1
82-8	49.4	-18	2	3.4
82-9	53.1	-25	High	42.7
82-10	55.9	-25	2	3.4

These results, while somewhat inconsistent, would seem to indicate that it is not the AsF₃ that is preventing the stage 2 compounds from achieving a resistivity lower than $3\mu\Omega\text{ cm}$.

B - Antimony Pentafluoride - Graphite Cored Wires

Attempts have been made at finding applications for the enhanced electrical properties that graphite gains upon intercalation. One of these has been in the production of swaged composite wires consisting of an intercalated graphite core encased in a metal sheath. This most recent work dealt with SbF_5 as the intercalate and copper as the sheath material. The purpose is to end up with a wire which conducts electricity better than conventional conductors, but is lighter.

It should be stated at the onset that this effort did not yield the desired results for reasons that will be explained. What follows is a description of those procedures that were tried which will be useful as a reference for any future work on this problem.

I - Procedures

The single largest problem in the preparation of swaged composite wires is the occurrence of cracks and fissures in the copper sheath which develop during the swaging process. This exposes the air-sensitive SbF_5 intercalated graphite, raising the resistance. Unfortunately, those wires which were successfully swaged to a sufficiently small size showed resistivities, when measured with a Kelvin bridge, higher than that for a pure copper wire.

a) Ampoule Geometry

The first task was to find a metal geometry which at least tended to minimize the cracking problem during swaging. This consisted mainly of varying the ID to OD ratio in the copper ampoule. A smaller ratio meant less frequent splitting, but also meant that less graphite was being used in the composite. After many attempts, the configuration shown in Figure 2 was chosen. The ampoules made during this early work were machined out of stock copper rod. Once the geometry was decided upon, only OFHC copper rod was employed in order to maximize the conductivity of the composite.

In the initial work done in investigating the geometry issue, the ampoules were found to swage better if they were filled loosely with graphite rather than packing them full using a hammer and plunger. This method, called tap filling, involved letting the graphite settle by tapping the ampoule gently on the table as it is filled. Although less graphite was used, somewhat better success was achieved for the swaging process.

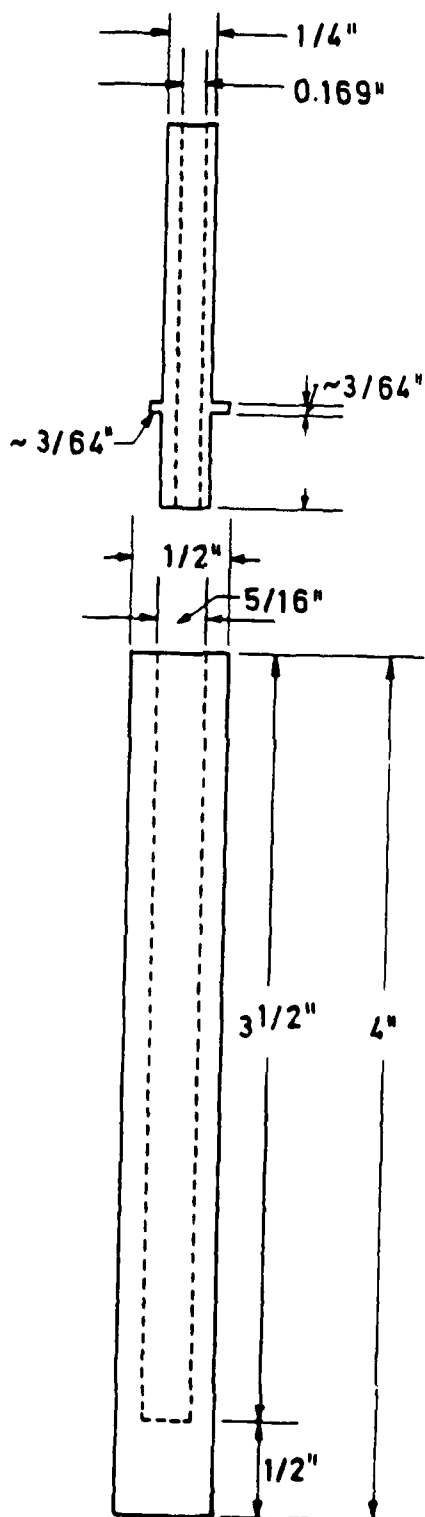


Figure 2. Copper ampoule used for preparation of swaged wires.

b) Filling Techniques

Numerous different methods were tried in the preparation of the ampoules for the swaging process. Some of these involved filling the ampoule with pristine graphite powder and then intercalating with SbF_5 . Others used pre-intercalated powders as the filler.

The first series of experiments attempted to have the intercalation proceed after the ampoules were filled with pristine graphite (Superior #2001). Initially, the caps were arc-welded to the ampoules after the ampoules were already filled with graphite. This proved difficult as the graphite would tend to interfere with the welding process, often leaving the seal with leaks. To alleviate this problem, the caps were welded first and filling was done later. This made it possible to leak check the ampoules before filling them with graphite.

After tap filling an ampoule (with typically about 2.5 gm of graphite), a small ball of fine copper wire is placed on top followed by some pyrex glass wool. These last two are used to prevent the graphite powder from being sucked into the vacuum line, but either or both are optional if caution is observed while evacuation is performed. The ampoule is connected to the apparatus shown in Figure 3. While being pumped on overnight, the ampoule is heated to about 150°C to remove any water in the system.

After the drying out of the graphite is completed, the ampoule is sealed off and attached to a graduated cylinder as in Figure 4. In a glove box, 1-2 ml of SbF_5 is added to the cylinder side, which is then capped with a stop-cock ground glass joint connector for removal from the glove box back to the vacuum line.

Now on the vacuum line, the cylinder side is evacuated of the glove box atmosphere and the SbF_5 is pumped on to remove unwanted absorbed gases. The cylinder and ampoule arrangement is sealed off from the vacuum line, the break-seal is broken, and the set-up is placed in an oven ($\sim 140^\circ\text{C}$). After baking for up to 600 hours in some cases, the ampoules were crimp-sealed just below the Swagelok connection. They were then swaged and when possible, measured for resistivity.

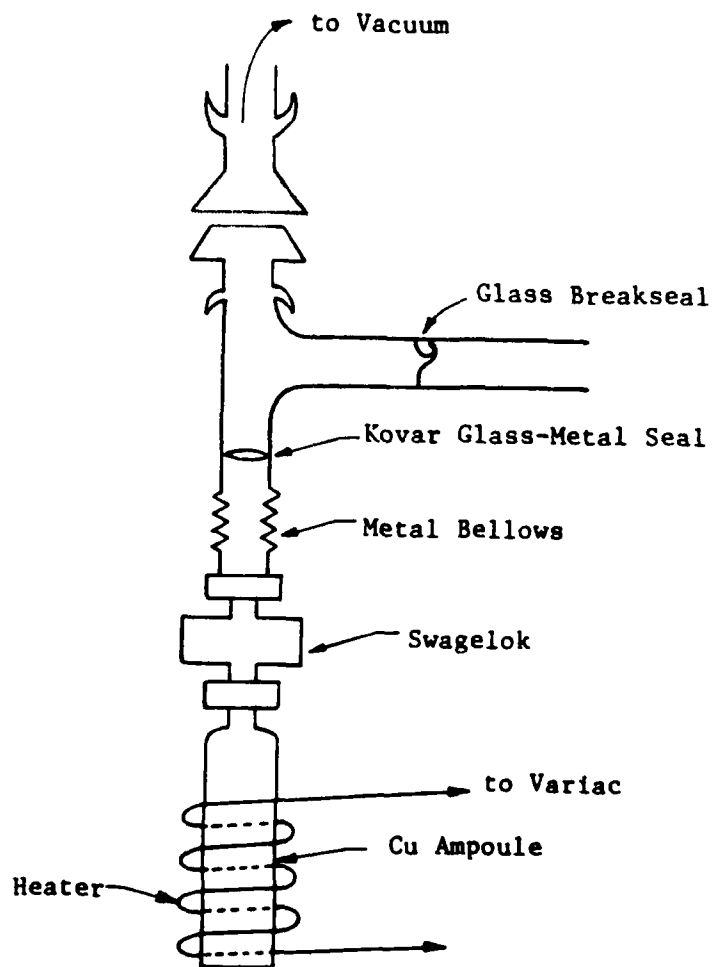


Figure 3. Apparatus for drying out graphite powder for swaged wires.

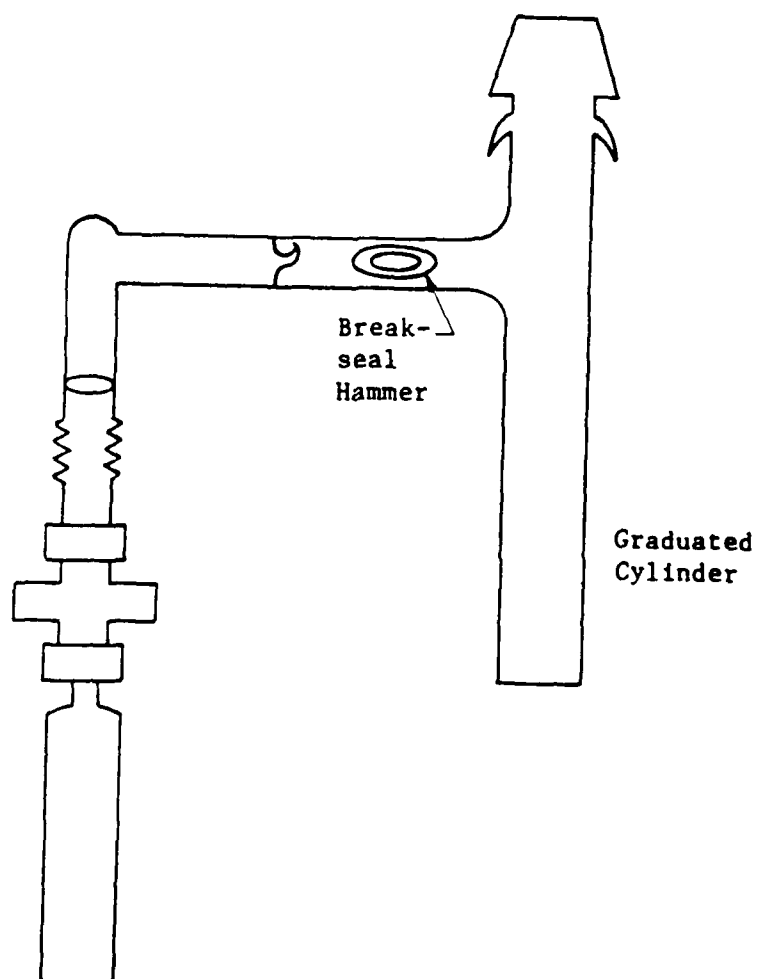


Figure 4. Apparatus for adding SbF_5 to graphite for swaged wires.

A second method involved half filling the ampoules with dried out graphite in a glove box. SbF_5 was then pipetted directly into the ampoules, followed by enough graphite to tap fill. The ampoules were sealed with a Swagelok plug, removed from the glove box, and baked at 150°C for about 45 hours. Different amounts of SbF_5 and graphite were used.

The third method utilized pre-intercalated SbF_5 graphite powders of various stage as the filling material. The ampoules were tap filled in a glove box and crimp-sealed for immediate swaging.

Other procedures involved attempts at reconstructing earlier work done by Gan (13). Ampoules of OFHC copper were machined as shown in Figure 2. Three different methods were employed.

- A. After filling an ampoule halfway with dried pristine graphite (Superior #2135) in an inert atmosphere glove box, enough SbF_5 (~ 0.3 ml) was pipetted in to yield a 50%-50% mixture by mass of graphite and SbF_5 . The rest of the graphite powder was added and then the ampoule was sealed. Sealing was done by adding some copper powder and closing the opening with a plug of solid Pb/Sn solder. The seal was secured by melting the solder in place with a hot soldering iron.
- B. After filling an ampoule halfway with Graphimet 50-50, 5 drops of SbF_5 (~ 0.02 ml) were added before the rest of the graphite based mixture was included. The ampoule was sealed as in A.
- C. After filling an ampoule halfway with Graphimet 50-50, ~ 0.15 ml of SbF_5 was added, followed by the rest of the Graphimet. The ampoule was sealed as in A.

2 - Results

The problems of cracking and splitting of the wires during swaging remained the larger problem. Less than 40% of the wires were undamaged after swaging from 0.5" OD to 0.125" OD. Annealing the Cu ampoules at 850°C for 1 hour before filling did not reduce the cracking problems.

Although most of the wires showed low resistivities, averaging about $1.9 \mu\Omega\text{cm}$, this could very well be due almost entirely to the copper sheath conductivity. Chemical analysis of wires of 0.125" OD showed that 98-99.5% of the wire mass is copper. There is a graphite core after swaging as can be seen by dissolving away the sheath in HCl or HNO₃. The procedures for the analysis of Sb content in the wires were very unreliable.

III. CONCLUSION

The objectives of this program, confirmation of the previously reported high electrical conductivity in arsenic pentafluoride and antimony pentafluoride intercalated graphites, were not accomplished. That is, with the materials and procedures used which were chosen to replicate those of previously reported high conductivities did not produce a similar result. It is therefore concluded, that while intercalated graphite with arsenic pentafluoride or antimony pentafluoride increases its electrical conductivity by very large amounts, other unknown and presently uncontrolled material factors limit access to the highest values. Understanding of these unknown factors awaits the right experiment.

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V. ADDENDUM

Review of Japanese work confirming ultra high electrical conductivity in arsenic pentafluoride intercalated graphite.

Following the initial reports from the University of Pennsylvania by Vogel (2) and Vogel and Gan (3) that high electrical conductivity could be obtained by intercalating graphite with antimony pentafluoride, and by Foley, Zeller, Falardeau and Vogel that similar results could be obtained with arsenic pentafluoride in graphite, there was a flurry of activity to replicate these exciting results. This activity occurred in laboratories around the world and was responsible for a large spurt of research effort involving two dimensional graphite intercalation compounds. However, although many laboratories succeeded in producing large increases in electrical conductivity parallel to the hexagonal planes in graphite, no one was able to reproduce the original result of electrical conductivity greater than copper or silver.

At the NATO Advanced Research Institute on Molecular Metals at Les Arcs, France, September 10-16, 1978, Vogel and Zeller (14) asserted that the chemical and physical perfection of the host graphite lattice has an important effect on this scattering of carriers. Thus, with any given chemical system, say arsenic or antimony pentafluoride intercalated graphite of a certain composition, it is the chemical and physical perfection of the graphite that determines how high the electrical conductivity can be.

Now, all of the attempts to replicate the high electrical conductivity of arsenic pentafluoride intercalated graphite used HOPG (Highly Oriented Pyrolytic Graphite) as the host material, depending on the generosity of A. W. Moore (8) and the Union Carbide Corporation (9). This material, as Moore frequently emphasized in private discussions, was developed for other purposes than providing high in-plane electrical conductivity.

Following in the directions pointed by the Vogel and Zeller paper, Shioya, Matsubara and Murakami (15) and Matsubara, Yamaguchi, Shioya and Murakami (16) intercalated arsenic pentafluoride into vapor grown fibers and films respectively to obtain the high electrical conductivity reported originally by the group at University of Pennsylvania. The Japanese workers used vapor grown fibers and films prepared by the decomposition of benzene followed by heat treatment at 3300°C using the methods developed by Koyama and Endo (17) but with somewhat higher temperatures of anneal. The benzene precursor material and the high annealing temperature lend themselves to the production of a graphite host material with the desired high chemical and physical perfection. Apparently the HOPG

material used in the original experiments done by the University of Pennsylvania group fortuitously had favorable chemical and physical perfection.

The Vogel and Zeller paper (14) suggested that electrical conductivities 10 times the value for copper may be obtained from graphite intercalated with strong acids. Now that the higher than copper conductivity has been experimentally confirmed and effect of the graphite host lattice perfection has been brought to a state of better understanding, it is time to proceed with the research goal of ultra high electrically conducting graphite intercalation compounds.

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